NIST Combinatorial Methods Center

FOCUSED PROJECTS
NON-PROPRIETARY RESEARCH AGREEMENT
APPENDIX A – HIGH THROUGHPUT METHODS FOR THE EVALUATION OF ADHESIVE PERFORMANCE

Article 1. **INTRODUCTION**

A wide variety of PSA's and epoxy resins are used in industrial and commercial applications. PSA performance is strongly dependent on the nature of the adhesive base, key formulation components, environmental parameters, substrate parameters and other specific factors. Likewise, a thermally curing epoxy resin's performance is affected by composition (epoxy resin vs. curing agent), pre and post curing temperature and rate, substrate surface characteristics, and other specific factors. This makes the development of a high throughput approach for the evaluation of PSA's and epoxies a challenging task. This project is directed towards developing a testing framework that will be used to evaluate adhesive and epoxy resin performance as a function of key manufacturing and environmental variables. This focused project aims to develop a high-throughput test method that will quickly characterize and evaluate adhesive and epoxy resin performance, correlate performance to measured adhesive and epoxy characteristics and provide sufficient experimental information to facilitate selective in-depth study of these systems.

Article 2. **BACKGROUND**

Peel and tack tests are often used to measure the performance of an adhesive. The former is a more industrially relevant qualitative measure of pressure sensitive adhesive performance [1] and the latter has recently emerged as a potential method to better characterize and visualize the mechanisms occurring during the debonding of a soft adhesive layer from either a flat or spherical probe [2,3]. Each test represents a slightly different approach. The peel test is a qualitative method that measures the maximum force required to separate an adhesive strip from a substrate. It allows one to quickly compare the performance between different adhesives and is a valuable tool for industrial testing. Tack tests not only measure the force exerted by the adhesive on the probe during debonding, but also the adhesive-probe contact area and the displacement required to completely separate the probe from the adhesive. A semi-quantitative test that quickly determines relative or absolute adhesive performance, but also permits further investigation of adhesive debonding mechanisms would be a unique and powerful tool for adhesive investigations. Wedge tests are useful to probe interfacial debonding at glassy interfaces (e.g. the fully cured epoxy interface). A sharp wedge such as a razor blade is driven into the interface at a known velocity and the crack propagation front is imaged to determine the work of adhesion and interfacial energy, given the modulus of the two materials is known.

Combinatorial methods utilize high-throughput measurement techniques to investigate a multi-variant parameter space more efficiently. The NIST combinatorial methods center (NCMC) has been actively developing combinatorial approaches to probe polymer adhesion. One such approach builds on the contact mechanics proposed by Johnson, Kendall, and Roberts (JKR), where the adhesion of a spherical indenter to a substrate is ascertained by following the contact area of the indenter during a loading and unloading cycle. The multi-lens combinatorial adhesion test (MCAT) is a high-throughput adhesion test bed currently under development at the NCMC. This high-throughput adhesion test employs an array of hemispherical lenses attached to a vertical actuator to perform from several hundred to several thousand-probe tack tests at one time. When these lenses are brought into contact with a gradient library, adhesion is measured over a large parameter space. The MCAT geometry is extremely flexible, allowing investigation of several different types of materials. The lens array may be fabricated from a material as rigid as glass, fully or partially cured epoxy or even an elastomer such as polydimethylsiloxane. The surface of the array may be chemically modified with a monolayer or coated with a thin film of polymer [4]. The MCAT measurable variables from each lens include the lens displacement (δ), the contact area (a). Potentially the load (P) applied to each lens, within the lens array, is measurable, although not in the present MCAT set-up. To first order, from this information, relevant measures of adhesive performance can be evaluated knowing the maximum load (P_{max}) achieved during unloading, and the total displacement (δ_{total}) to detach each lens on the array from the adhesive. The freedom to change MCAT geometry ensures the applicability of this technique to a large number of adhesive-substrate systems.

The current MCAT geometry is well suited for the measurement of adhesive forces across partial to fully cured epoxy resins, but is unsuited for investigation of soft materials such as PSA's or uncured epoxy films. In order to better understand the debonding process for the soft materials, the current lens array will require modification from an array of hemispherical lenses to an array of posts. The post array ensures constant contact area between the probe and adhesive layer, which simplifies the stress-strain analysis required to quantify the debonding process. The array of posts may be created using developed soft lithography techniques and modified using techniques perfected utilizing the current hemispherical lens arrays in use with the MCAT technique.

References:

1. Satas D. Handbook of pressure-sensitive adhesive technology, Van Nostrand Reinhold, New York, 1982.
2. Creton C.; Fabre P. *TACK* Comprehensive Adhesion Science, (II) 2002.
3. Shull K. R.; Ahn D.; Chen W. L.; Flanigan C. M.; Crosby A. J. *Axisymmetric adhesion tests of soft materials*, Macromolecular Chemistry and Physics (199) 1998, 489-511.
4. Crosby A. J.; Karim A.; Amis E. J. *Combinatorial investigations of interfacial failure*, Journal of Polymer Science Part B: Polymer Physics (41) 2003, 883-891.
5. Chuang HK; Chiu C.; Paniagua R. *Avery Adhesive Test yields more performance data than traditional probe*, Adhesives Age (10) 1997, 18-23.

Article 4. **COLLABORATION AND DISSEMINATION**

A meeting will be held six weeks after the formal launch of the project as well as at six-month intervals for the duration of the project. Quarterly reports will be submitted to the members with updates more frequently via conference calls and other postings. In order to facilitate the collaboration, specifications for methods, instruments, programs, data analysis, and other aspects of this work will be available to members during the course of the project. A summary report will be provided within two months of the end of the project. The NCMC labs will be open to prearranged visits from member scientists interested in hands-on participation in method development.

As with base level membership in the NCMC, all of the research carried out in the Focused Project is non-proprietary and is intended for publication in the public domain. No proprietary information or materials will be solicited or accepted by NIST from member organizations. The scope of the work by NIST included in this focused project is limited as described in Article 5 below.

Article 5. **PROJECT MILESTONES**

5.1 **First year:**

5.1.1 Select a suitable model adhesive-tackifier blend and a model epoxy system and establish techniques for library generation of identified parameters.

5.1.2 Conduct preliminary tests on at least one selected model PSA and one selected epoxy system.

5.2 **Second year**

5.2.1 Develop a systematic process for library generation, characterization and testing of adhesive and epoxy films and integration with the (NCMC) informatics database.

5.2 **Second year cont'd.**

5.2.2 Correlate properties with parameters to develop understanding of cause-effect relationships for the model PSA and epoxy system (s) studied and draft summary report on each.

5.2.3 Assist focused project members investigation of a suitable non-proprietary commercial blend system to determine cause-effect relationships between parameters and performance and provide feedback for optimization.

Article 6. **FINANCIAL OBLIGATION**

Member's project fees payable to NIST are set at $20,000 per year.